

## STUDIES OF TRANSFORMATIONS OF INORGANIC CONSTITUENTS IN A TEXAS LIGNITE DURING COMBUSTION

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### INTRODUCTION

The effects of coal composition and combustion conditions on the mechanisms of ash formation are being investigated at the Energy and Mineral Research Center (EMRC). The objective of the study is to develop a unified understanding of formation of ash components during pulverized coal combustion. This understanding will aid in predicting the formation of ash deposits, erosion of boiler parts and formation of fine particulate that is difficult to collect.

The approach used to obtain a better understanding of inorganic transformations that take place during combustion of pulverized coals involves the following key elements: 1) an effective multidisciplinary means of characterizing the original coal for its inorganic content; 2) a controlled, small-scale combustion regime that can be quickly and easily manipulated to simulate the time and temperature profile in a full scale utility boiler; and 3) an effective means of characterizing the combustion products, such as fly ash generated in the small scale combustor. The abundance and mode of occurrence of inorganic components in coal are quantified using a combination of computer controlled scanning electron spectroscopy (CCSEM) and chemical fractionation techniques<sup>1,2,3,4</sup>. The CCSEM is used to determine the size and abundance of mineral grains in the coals. Chemical fractionation is used to quantify the abundance of organically associated elements. Ash is produced under carefully controlled combustion conditions using an entrained flow reaction or drop tube furnace system which simulates full scale utility boiler regimes<sup>5,6,7,8</sup>. The resulting ash is collected in a multicyclone which size-classifies the ash. The size segregated ash is characterized using a scanning electron microscope.

There are many methods of ash deposit and fly ash characterization which greatly enhance the ability of researchers to determine, with reasonable accuracy, the size distribution and composition of fly ash<sup>9,10,11,12</sup>. This allows for inferences to be made concerning the crucial factors involved in the transformation of inorganic components and also allows for the direct comparison of original mineral material with fly ash. In this particular study the mechanisms of coal ash formation were investigated for Monticello lignite from Titus County, Texas. The Monticello lignite was carefully prepared, analyzed, and combusted. The resulting ash was analyzed and compared to the original inorganic components in the coal.

### EXPERIMENTAL

#### Coal Characterization

Monticello coal was characterized using a variety of analytical techniques. Standard ASTM coal and coal ash analyses were performed. Chemical fractionation<sup>1</sup> and computer-controlled scanning electron microscopy (CCSEM)<sup>6,8</sup>

were used to ascertain abundance and distribution of inorganic components, as well as the size and type of mineral grains in the coal.

Chemical fractionation was used to selectively extract elements from the coal based on solubility, which reflects their association in the coal. Briefly, the technique involves extracting the coal with water to remove water-soluble elements. This is followed by extraction with 1M ammonium acetate to remove elements that are associated as salts of organic acids. The residue of ammonium acetate extraction is then extracted with 1M HCl to remove acid-soluble species in the form of hydroxides, oxides, carbonates, and organically coordinated species. The components which remain in the residue after all three extractions are assumed to be associated with the insoluble mineral species such as clays, quartz, and pyrite.

The CCSEM analysis was performed on a 53-74 $\mu$ m sized fraction of Monticello lignite. Approximately 1000 mineral grains were type-classified based on elemental compositions, and sized according to average diameter. The size categorization of the grains was selected to be consistent with multicyclone cutpoints used in size-classifying the fly ash produced from this same coal fraction in the drop tube furnace. The CCSEM technique tends to underestimate the true average diameters of the mineral grains. Since the coal sample that is analyzed is a cross-section, the mineral grains observed are also cross-sectioned. This means that a mineral that has been sliced exactly at its average diameter is rare. To verify the CCSEM sizing technique, the coal minerals were also sized using a coulter counter analysis of a low temperature ash sample.

#### Fly Ash Production and Characterization

Fly ash was produced using an entrained-flow tube furnace, also known as a drop-tube furnace. The drop-tube furnace is a laboratory scale furnace system that has the ability to mimic conditions of commercial combustors without the high cost associated with pilot-scale combustion testing. The combustion temperature, residence time, and gas cooling rate can be closely controlled and monitored. Table 1 gives the combustion parameters used to produce the ash. More specific details of the EMRC drop-tube furnace are described elsewhere<sup>3</sup>. Fly ash was cooled by means of a fly ash quenching probe and collected using a multicyclone. The multicyclone aerodynamically separates the fly ash into 6 stages or aerodynamic categories. Each of the fly ash samples collected from the 6 stages was mounted in a suitable SEM mount and characterized using scanning electron microscopy and electron microprobe analysis. A technique called scanning electron microscopy point (SEMP), which was developed at the EMRC, was employed to determine the relative abundance of phases present in the fly ash particles. The types of phases identified include: (1) phases that resemble the original components in the coal, (2) phases that have molar and weight ratios of elements that are consistent with known crystalline species, and (3) unclassified species that do not fit in the first two categories. This information is used to compare not only the compositional changes in the ash, but also the changes in phases as a function of particle size. Specific information regarding the SEMPC technique can be found elsewhere<sup>10</sup>.

## RESULTS AND DISCUSSION

### Coal Characterization

The chemical fractionation results for the Monticello coal are found in Table 2. The large percentage of aluminum, silicon, potassium, and titanium remaining after the extraction implies that these elements were mostly associated with insoluble minerals as clays, quartz, and possibly rutile (titanium oxide). The results suggest that significant amounts of the alkali and alkaline earth elements are associated in the coal as salts of organic acid groups. For example, most of the magnesium, calcium, and strontium were removed by the ammonium acetate extraction. Potassium appears to be associated with a clay mineral. The iron was distributed as roughly half acid-insoluble sulfides (probably pyrite) and half organically coordinated or acid soluble minerals.

The Monticello coal has a relatively high ash content of 15%. From the chemical fractionation analysis it was calculated that at least 77.8% of the ash or 11.7% of the coal consisted of discrete mineral phases. Table 3 shows the distribution of discrete mineral phases in the Monticello 53-74 $\mu$ m coal, as determined by CCSEM. Nine minerals were observed in this size fraction of Monticello coal including: quartz, kaolinite, Fe-aluminosilicate, K-aluminosilicate, Ca-aluminosilicate, iron oxide, pyrite, rutile, and an aluminosilicate-gypsum mixture. The Fe and Ca-aluminosilicates were probably a type of montmorillonite and the K-aluminosilicate was most likely illite. One limitation of the CCSEM technique is that it is difficult to accurately classify clay minerals, except for kaolinite and in some instances, illite. Quartz was the most abundant mineral and was the dominant phase in all of the size ranges except for the >8.0-11.0 $\mu$ m range where kaolinite was the major phase. Figure 1 shows the abundance of five important mineral types observed in the six size categories. Quartz and Ca-aluminosilicate (montmorillonite) show approximately 70% of their mass in the >11 $\mu$ m size range. All five minerals shown in Figure 1 have a slight increase in content in the 2.1-4.4 $\mu$ m size range. Kaolinite is evenly distributed in a 1.2-11.0 $\mu$ m size range.

### Fly Ash Characterization

The results of the SEMPC analyses are listed Table 4. Hauyne, which is a sodium - calcium aluminosilicate with sulfur, was observed in all of the samples except the 2.1-4.4 $\mu$ m range. The highest content of hauyne was observed in the 1.2-2.1 $\mu$ m range. Calcium oxide (which refers to species such as CaCO<sub>3</sub> and CaO), montmorillonite-derived material, illite, and iron oxide were detected only in small amounts in all stages. All size categories contained significant quantities of kaolinite-derived materials with the highest level found in the 1.2-2.1 $\mu$ m range. Anhydrite was relatively enriched in the 8.0-11.0 $\mu$ m size fraction and on the final filter. Plagioclase in the form of solid solution phases between end members albite (NaAlSi<sub>3</sub>O<sub>8</sub>) and anorthite (CaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub>), was observed at low levels, with none observed in the sample from the 4.4-8.0  $\mu$ m size categories. The quartz content of the samples varied markedly between size categories. In the >11 $\mu$ m and 4.4-8.0 $\mu$ m size fractions, 25.4 and 31.8% quartz were detected, respectively. Melilite was present in all of the multicyclone stages except for the 2.1-4.4 $\mu$ m size range. The sample from the 4.4-8.0 $\mu$ m size category had the greatest number of unidentified phases (45.5%), whereas the 8.0-11.0 $\mu$ m and 2.1-4.4 $\mu$ m size ranges had the fewest unidentified phases (63.5 and 65.4%, respectively).

The chemical compositions (on an  $\text{SO}_3$ -free basis) of each sample are listed in Table 4 along with the Si/Al molar ratio. The most striking result shown in Table 4 was the high level of Mg and Fe in the ash collected on the final filter. The very high Fe and Mg contents in the final filter suggests they may have vaporized and subsequently condensed to form submicron size particles, or that they were organically associated which can also produce very small particles during combustion. The chemical fractionation results suggest that approximately 53% of the iron is associated organically and/or as a carbonate mineral. The CCSEM data shows that a very low level of iron was observed in the form of iron oxide/carbonate minerals. In addition, no significant amount of pyrite was found.

The Si/Al molar ratios did not indicate any apparent trends between the multicyclone samples. The 4.4-8.0 size range had the highest value while the 1.2-2.1  $\mu\text{m}$  range had the lowest. The data indicated that the  $>11\mu\text{m}$ , 4.4-8.0  $\mu\text{m}$  ranges were relatively enriched with silica (presumably quartz), whereas the 8.0-11.0  $\mu\text{m}$  and 1.2-2.1  $\mu\text{m}$  ranges were relatively depleted in silica.

The particle size distributions of the Monticello coal minerals, fly ash, and low temperature ash (LTA), as determined by CCSEM, multicyclone, and coulter counter analysis respectively, are shown in Figure 2. In all cases the majority of the mass was present in the larger size ranges. The minerals as determined by CCSEM show a bimodal distribution concentrated at the 2.1-4.4  $\mu\text{m}$  and  $>11\mu\text{m}$  ranges. The CCSEM technique tends to underestimate the average diameter of mineral grains in cross-section. The LTA distribution shows nearly the same amount of material at the 2.1-4.4  $\mu\text{m}$  range as the CCSEM distribution, however, it has more particles with sizes in the 4.4-11.0  $\mu\text{m}$  range and less particles in the  $>11\mu\text{m}$  range. Two possible explanations for the differences between the LTA and CCSEM distributions are: 1) clay aggregates, which may be counted as only one particle in the CCSEM analysis, are usually broken up into smaller particles during the low temperature ashing process and 2) the LTA samples include mass from organically bound elements which are not quantified by CCSEM.

Figure 2 also shows that the fly ash distribution has very little mass in the lower size categories, but most of the particles are  $>11\mu\text{m}$ . This is evidence for the process of coalescence and expansion whereby variously sized minerals and organically bound inorganics in a coal particle coalesce to form a larger fly ash particle. Further expansion may be due to the escape of gaseous or volatile matter.

Comparisons were made between the content of selected minerals in the original coal and their corresponding phases in the fly ash on a percent mass basis. The Monticello coal ash concentration was determined to be 15% on a dry basis. From the chemical fractionation analysis it was calculated that at least 78% of the ash or 12% of the coal consisted of true mineral phases (referred to as mineral matter here). When comparing the percentage of coal mineral phases to the inorganic phases present in the fly ash it must be remembered that the fly ash consists of both organically bound elements and mineral matter. The CCSEM analysis of coal minerals does not include the minute, organically bound inorganics. Therefore, to compare the coal mineral and fly ash data, the mineral data was multiplied by a correction factor of 0.78 to evenly weight the comparisons. The limits of experimental error for this comparison procedure are approximately 10-20%.

Comparison of the percent kaolinite in the coal to the percent kaolinite-derived in the fly ash revealed that the concentrations for corresponding size ranges were within 15% of each other except for the 8.0-11.0 $\mu$ m range (Figure 3). Kaolinite in the coal had lower concentration than the kaolinite-derived in the fly ash, excluding the <1.2 $\mu$ m and 8.0-11.0 $\mu$ m ranges. This suggests that a large percentage of the finer grained kaolinite in the coal collects extraneous inorganic matter during combustion which thereby increases its mass to a higher value than what was seen in the original coal. Sodium, magnesium, and calcium, most likely from organically bound cations, were the primary additions to the kaolinite-derived mass. The distribution of quartz, as shown in Figure 4, revealed a higher percentage of quartz in the coal than in the fly ash for all size ranges. Both curves have the same trends of slope when lines are drawn between the size ranges.

### CONCLUSIONS

The Monticello 53-74 $\mu$ m coal and its corresponding drop tube furnace fly ash were characterized for their inorganic matter as a means of better understanding the inorganic transformations involved. The Monticello coal had a high ash content of 15% and approximately 78% of the inorganic constituents were determined to be mineral grains. The major mineral types identified included quartz, kaolinite, and Ca-aluminosilicate (montmorillonite). Major organically bound elements included sodium, magnesium, calcium, strontium, and barium. Iron content in the Monticello coal consisted of roughly half insoluble sulfides (pyrite) and half organically-coordinated or other acid soluble species. Although the CCSEM technique provides valuable data concerning the size and distribution of mineral phases, in its current stage of development the CCSEM technique appears to underestimate the total mass of minerals present. The CCSEM technique is best applied along with chemical fractionation; the combination of these two techniques provides a very complete picture of the form and nature of inorganic constituents present in a low-rank coal.

The particle size distribution of the Monticello minerals, as determined by CCSEM, had a bimodal distribution with over 60% of its mass >11 $\mu$ m. The LTA distribution had only 33% of its mass >11 $\mu$ m. The differences between the LTA and CCSEM distributions may be due to the breakup of clay aggregates during the low temperature ashing or the exclusion of organically bound elements in the CCSEM analysis. The size distribution of the fly ash showed 90.9% of its mass >11 $\mu$ m. Apparently, smaller mineral grains and organically bound inorganics are agglomerating or being assimilated into the melt phase of other material during combustion.

Analysis of the 6 multicyclone size fractions of fly ash using SEMPC identified 13 types of inorganic phases. The most abundant phases observed were hauyne, kaolinite, anhydrite, plagioclase, quartz, melilite, and unclassified material. Hauyne, a complex aluminosilicate containing sulfate, was mostly concentrated in the smaller size fractions. Kaolinite-derived phases had a fairly uniform distribution in all of the size ranges except for an anomalously low value in the final filter. The majority of the anhydrite was found in the final filter fraction probably due to deposition as gaseous or very fine particulate matter. Quartz content varied markedly between size ranges. Higher levels of Mg and Fe were noted in the smallest size fraction (<1.2 $\mu$ m). These values may be a result of how the Mg and Fe was associated in the coal; as a significant portion of both Mg and Fe are associated with the

organic fraction of the coal. Therefore, the relative sizes of the resulting ash particles containing these elements may be limited to a finer size fraction.

In comparing coal minerals to fly ash minerals and phases on a percent mass basis, several trends were noted. A large percentage of the finer-grained kaolinite in the coal appears to have combined with other minerals or extraneous inorganics during combustion. This was evidenced by the increase of non-crystalline kaolinite (kaolinite-derived) in the fly ash as compared to the original coal.

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TABLE 1  
DROP-TUBE FURNACE RUN CONDITIONS

<u>Monticello</u>	
Particle Size	53-74 $\mu\text{m}$
Primary Air	1.25 L/min
Sec. Air	4 L/min
Vacuum	15 L/min
Quench Gas	3 L/min
<u>Temperatures</u>	
Preheat Injector	1000°C
Furnace 1	
In	1500°C
Out	1315°C
Furnace 2	
Mid-Point	1280°C
Residence time	sec.

TABLE 2  
CHEMICAL FRACTIONATION RESULTS FOR  
MONTICELLO COAL

	<u>Initial</u> <u>(<math>\mu\text{g/g}</math> dry coal)</u>	<u>% Removed</u> <u>by <math>\text{H}_2\text{O}</math></u>	<u>% Removed</u> <u>by <math>\text{NH}_4\text{OAc}</math></u>	<u>% Removed</u> <u>by <math>\text{HCl}</math></u>	<u>%</u> <u>Remaining</u>
Na	315	31	31	0	18
Mg	1180	0	70	4	26
Al	9110	0	0	5	95
Si	23600	0	0	1	99
K	335	21	5	0	74
Ca	7320	0	77	13	10
Ti	395	0	0	15	85
Fe	2200	0	0	53	47
Sr	130	0	72	11	17
Ba	100	0	52	26	22



TABLE 3

CCSEM MINERAL COMPOSITION OF MONTICELLO COAL  
(Weight Percent of Total Discrete Minerals)

Mineral	Size Categories (micrometers)						Total % Minerals	Total Wt% Minerals (Coal Basis)
	<1.2	1.2-2.1	2.1-4.4	4.4-8.0	8.0-11.0	>11.0		
Quartz	0.39	2.82	10.47	8.16	2.09	55.41	79.35	4.34
Kaolinite	0.11	0.61	2.13	1.6	2.65	0.0	7.09	0.39
Fe-Aluminosilicate	0.02	0.14	0.25	0.0	0.0	0.0	1.40	0.02
K-Aluminosilicate	0.03	0.27	1.26	1.05	0.0	0.0	3.04	0.17
Ca-Aluminosilicate	0.14	0.58	0.68	0.0	3.42	3.42	4.82	0.26
Iron Oxide/Carbonate	0.0	0.10	0.10	0.0	0.0	0.0	0.9	0.01
Rutile	0.03	0.12	0.32	0.0	0.0	0.0	0.48	0.03
Alum-Silic/Gypsum	0.03	0.13	0.07	0.37	0.0	2.73	3.34	0.18
Pyrite	0.0	0.06	0.07	0.0	0.0	0.0	0.13	0.01
Gypsum	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00
Unknown	0.0	0.02	0.35	0.21	0.47	0.0	1.05	0.06
Coal								94.53
Total	2.75	4.89	15.77	11.40	5.63	61.56	100.0	100.00

TABLE 4  
PHASES DETECTED BY SEMPC FOR MONTICELLO FLY ASH

	<u>Filter</u>	<u>Stage 5</u>	<u>Stage 4</u>	<u>Stage 3</u>	<u>Stage 2</u>	<u>Stage 1</u>
Particle Size Cutpoints (μm)	<1.2	1.2-2.1	2.1-4.4	4.4-8.0	8.0-11.0	>11.0
Percent Mass	2.0	2.4	1.1	1.6	2.0	90.9
<u>Phase (Number %)</u>						
Hauyne	1.6	11.2	1.0	0.0	2.9	0.5
Calcium Oxide	0.0	0.0	0.0	0.0	0.0	0.5
Montmorillonite +	0.0	0.7	1.0	0.0	0.0	1.5
Calcium Silicate	0.0	0.7	1.0	0.0	0.0	0.0
Illite	0.0	0.0	0.0	0.0	0.6	0.0
Iron Oxide	0.0	0.0	1.0	0.0	1.7	0.0
Kaolinite +	1.2	22.4	13.0	18.2	16.6	12.2
Anhydrite	28.0	1.4	0.0	2.3	6.3	0.5
Plagioclase	0.0	1.4	1.0	0.0	1.2	1.0
Quartz	0.0	2.1	16.3	31.8	6.3	25.4
Melilite	0.4	2.8	0.0	2.3	1.1	2.0
Pyroxene	0.8	0.0	0.0	0.0	0.0	0.0
Unclassified	67.9	56.6	65.4	45.5	63.5	56.3
<u>Average Composition, wt% (SO<sub>3</sub>-Free)</u>						
SiO <sub>2</sub>	29.5	37.7	51.5	51.7	43.5	64.0
Al <sub>2</sub> O <sub>3</sub>	10.2	18.2	15.2	11.6	17.5	15.1
Fe <sub>2</sub> O <sub>3</sub>	26.0	6.7	5.7	4.6	5.8	2.6
TiO <sub>2</sub>	0.9	2.1	1.5	2.6	1.9	1.7
P <sub>2</sub> O <sub>5</sub>	0.2	0.3	0.5	0.4	0.4	0.5
CaO	15.8	27.4	19.9	23.1	23.7	12.6
MgO	16.0	5.6	3.7	4.0	4.6	2.3
Na <sub>2</sub> O	1.1	1.6	1.7	1.6	2.0	0.8
K <sub>2</sub> O	0.1	0.3	0.3	0.3	0.4	0.4
(SO <sub>3</sub> )*	8.70	6.4	4.2	3.7	4.6	0.7
<u>Average Si/Al Molar Ratio</u>						
	2.5	1.8	2.9	3.8	2.1	3.6

\*SO<sub>3</sub> added for comparison purposes.

+derived phases-resemble the original mineral in the coal.

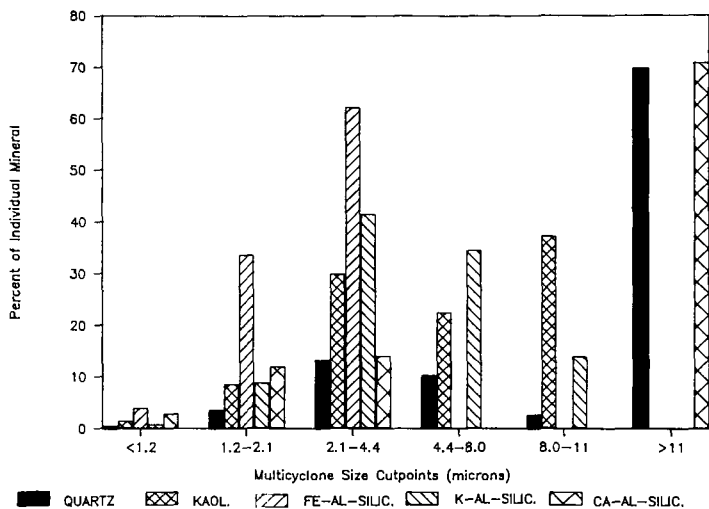


Figure 1. Size distribution of five individual mineral types in Monticello coal.

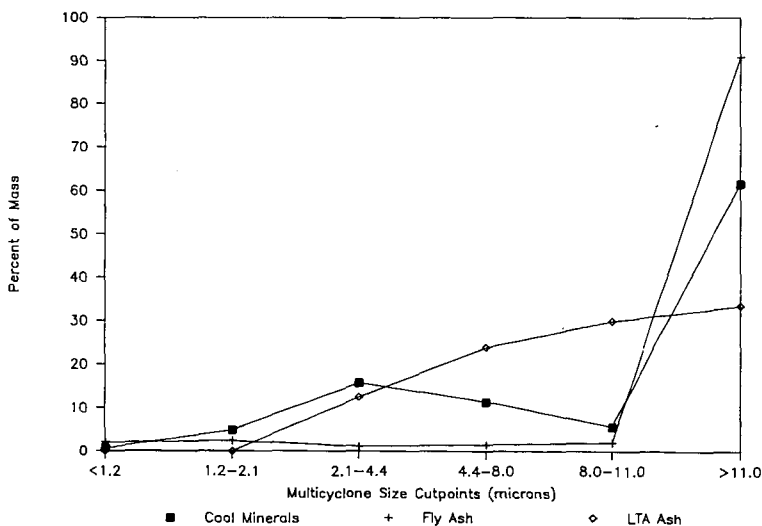


Figure 2. Particle size distribution of Monticello coal minerals, fly ash, and low temperature ash as determined by CCSEM, multicyclone, and coulter counter techniques, respectively.

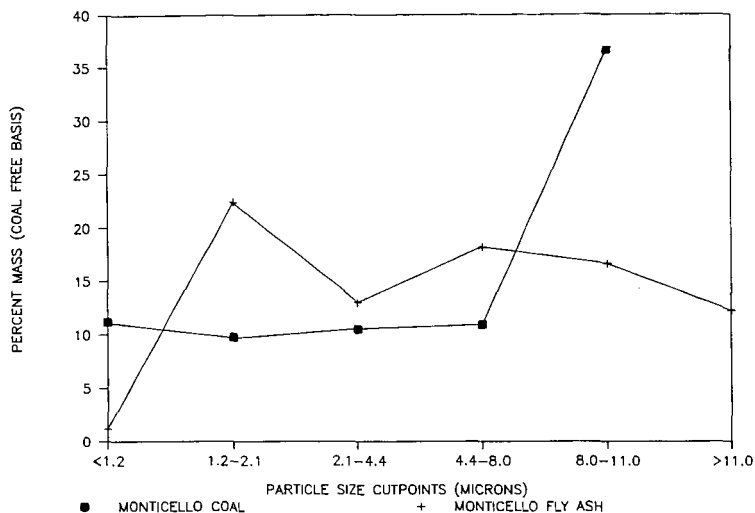


Figure 3. Distribution of Kaolinite in Monticello coal and Kaolinite-derived material in the Monticello Fly Ash.

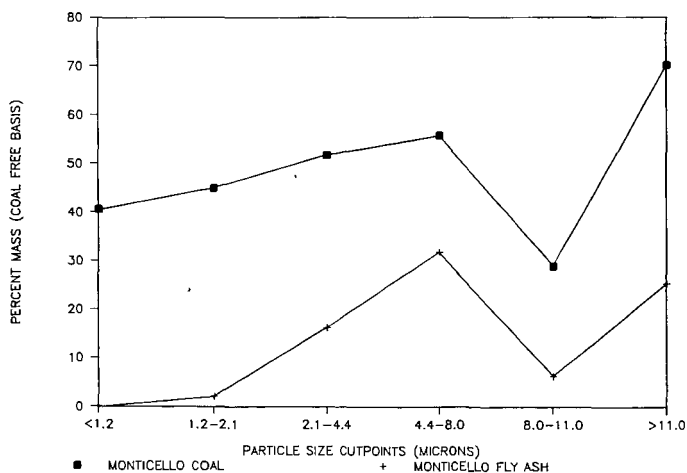


Figure 4. Distribution of Quartz in Monticello coal and Quartz-derived material in the Monticello Fly Ash.